HOT PRESSED 90+%Al$_2$O$_3$ CONCRETE

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In Partial Fulfilment of the Requirements
for the Degree of
MASTER OF TECHNOLOGY

by

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to the

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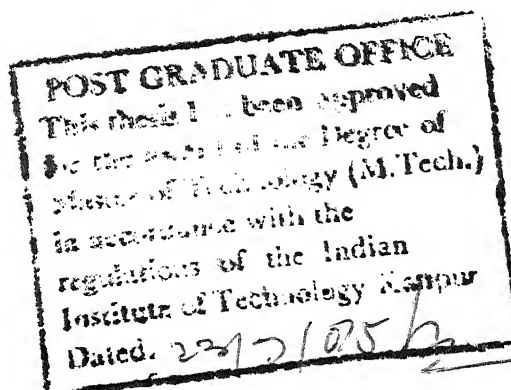INDIAN INSTITUTE OF TECHNOLOGY, KANPUR
JULY, 1985

CERTIFICATE

This is to certify that this work entitled ' HOT PRESSED 90+% Al_2O_3 CONCRETE', by Ajoy Kumar Bose has been carried out under my supervision and has not been submitted elsewhere for a degree .

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ABSTRACT

Fused alumina aggregates and high purity white high alumina cement (W.H.A.C.) were used to develop a concrete mix with 90+% Al_2O_3 content. Different compositions and process parameters e.g. cement content, water/ cement ratio and molding pressure were studied from point of view of strength.

Technique for hot pressing at 100°C was developed. This technique permitted a very low water/cement ratio resulting in a considerably higher strength values. The hot pressed bodies were compared with cold pressed cement bonded and phosphate bonded fused alumina bodies.

Maximum tensile strength upto 75 Kg/cm^2 could be attained (corresponding to = 800 Kg/cm^2 compressive strength) using 20% cement, 0.11 water/cement ratio and 1018 Kg/cm^2 molding pressure at 100°C, 10 minutes.

CHAPTER - I

INTRODUCTION

Prior to early sixties, the fire-resistance lining of industrial furnaces and similar industrial equipments operating at elevated temperature were constructed almost exclusively of precast and prefired refractories. Hence the different design, construction and operational considerations were invariably based on the prefired linings. A few exceptions were the in-situ products intended to use as morters, rammed conceretes, sprayed concrete and renderings for repair and similar surface treatments.

It is seemingly illogical to prefire refractories at their manufacturing stage when these are to be subjected to high operating temperature in usage. However in the traditional technology, prefiring of refractories, and even the rawmaterials is mandatory if the required properties are to be achieved and many difficulties relating to thermal behaviour, such as expansion, shrinkage and mechanical strength are to be avoided.

In recent years there has been a marked trend towards the elemination of the firing step in the manufacturing of refractories, thereby effecting economirs in

production costs and acquiring greater flexibility in
usage . This has been achieved by developing unfired
bricks or precast refractory components which possess
the necessary room temperature strength and load bearing
capacity at elevated temperature, while having adequate
dimensional stability. Typical examples are chemically
bonded basic refractories, concrete bonded with cements,
sodium-silicate [1] and castables bonded with magnesia,
phosphoric acid or phosphate bonds [28=32] . These permit
not only production of standard size bricks but also,large
size precast units for insitu constructions of monolithic
structures leading to many advantages over the conventional
methods in rationalisation, productivity, increased output
and economy.

Modern developments in process technology have
lead to increasingly severe furnace conditions. Two classes
of refractories have been developed to meet this challenge;
one, phosphate bonded refractory, and two, refrectory
concrete, specially 90+% Al_2O_3 concrete made with graded
fused alumina and high purity calcium-aluminate cement.

The cement bonded concrete has an advantage over
the phosphate bonded castables. The latter need to be cured
at above 300°C in order to develop a proper bonding, strength
and abrasion resistance.

This is a time consuming step specially in large construc-
tions. On the other hand cement bonded fused alumina
concretes, prepared with addition of organic fibers
to increase its permeability and or bonded with low cement
content and silica sol instead of water, can be heated
up to the operating temperature at a fast rate without the
danger of steam explosion. This new explosion and abra-
sion resistance concrete has been succesfully used in
[19],

    (i)   the discharge and cooler bends of large
          cement rotary kilns with planetory cooler.

    (ii) monolithic lances for gas and powder injec-
          tion used in laddle refining of steel.

    (iii) prefebricated blocks for lining of walking
          beam reheating furnances for billet and
          slabs in steel industries.

    (iv) lining of tapping spout of electric arc
          furnace.

    (v)   errosion resistance castable lining of
          refining vessels in petro-chemical industries,

where faster heat up schedule can save expensive down time
and eleminate risk for explosive spalling .

More over ceramic forming dies made with 90+%
Al_2O_3 have improved the production technology in hot

forming of stainless stee, titanium, and beryllium metal components in space age and air craft industries. These precision cast ceramic tools, dies, molds are also being used in the electronic industries [25].

High alumina ramming material in the center of electric arc furnace roof has resulted in longer life [26].

Concrete made of 45-90% Al_2O_3 with low, intermediate and high purity calcium- aluminate cements bond has been succesfully used in dry ash coal gassifier, where the temperatures encountered are 760-100°C, pressures 2.9-7.0 MPa and the atmosphere is saturated with steam [27].

Cement bonded alumina concretes are now begining to be used in India in increasing amount. The work reported here deals with finding optimal conditions for cold bonding of alumina with calcium -aluminate cement and for hot pressing of these bodies to make unfired preformed refractory and abrasion resistance ceramics.

CHAPTER -II

LITERATURE REVIEW

1. DEFINITIONS:

The word concrete is generally understood to mean a mixture of hydraulic setting cement, aggregates of different kinds and particle sizes and water, which hardens at the ambient temperature [1,2.3].

Heat resistance concrete is a type of concrete which retains its physical and mechanical properties within certain limits after long period at high temperatures [4].

The term ' cement ' means a finely ground hydralulic binding agent, consisting principally of CaO compounds with SiO_2, Al_2O_3 and Fe_2O_3, and which when mixed with aggregates and water sets by combination with water after a certain period and then continues to harden into a stonelike material, insoluble in and resistance to attack by water [5].

Although there are several classifications of high temperature concrete, our interest is in concretes of high strength, refractoriness (>1800°C) and abrasion

resistance made with high purity white high alumina cement and fused alumina aggregates.

The cement act as a hydraulic binding agent at normal temperature and it reacts with aggregates at high temperature giving rise to ceramic bond.

2. WHITE HIGH ALUMINA CEMENT:
A. COMPOSITION AND PHASES:

Composition of different hydraulic setting cements in shown in fig: II-1 in the SiO_2- Al_2O_3-CaO triaxial diagram. White high alumina cement contains 72-80% Al_2O_3 and rest CaO with minor impurities of SiO_2, iron oxide etc.

In the following discussion CaO, SiO_2, Fe_2O_3, H_2O are abbriviated as C,S,A and H respectively.

The major phases present in W.H.A.C. are, CaO, Al_2O_3 (CA) and CaO. $2Al_2O_3$ (CA_2) along with some amount of Al_2O_3 and 12CaO. $7Al_2O_3$ ($C_{12}A_7$) and traces of 2CaO. Fe_2O_3 (C_2F) and $2CaO.Al_2O_3.SiO_2$ (C_2AS), [6,7].

i) CA: It is white in color and has an alumina content of about 64.5% and melts at 1608^oC, [9]. It is a slow setting phase, particularly if rapidly quenched [8], but hardens with great rapidity after the final setting.

ii) CA$_2$: First thought to be C$_3$A$_5$, this compound was given the present formula by Tavasci and Lagergvist in 1973.   It contains 78.5% Al$_2$O$_3$ and has a fusion point of about 1770°C [9].   The pure compound reacts very slowly with water but hydration and strength development are accelerated in the solution of high pH values.   Once the hydration of CA$_2$ has started, the rate increases with increasing concentration of hydration product [10].

iii) C$_{12}$A$_7$: This is usually found in small amount in W.H.A.C.   Its amount in cement increases when the CaO/^{A}l$_2$O$_3$ ratio is increased. This compound hardens very rapidly. It has low fusion point.

iv) C$_2$AS: It has low fusion point and must be kept low in refractory grade W.H.A.C. It reacts extremely slowly with water, hence its presence in large amounts causes serious reduction in the rate of hardening of W.H.A.C.

v) C$_2$F: The percentage of Fe$_2$O$_3$ is kept very low in W.H.A.C., thus the amount of this phase present is negligible.

B. HYDRATION OF W.H.A.C. [11,12,13]:

CAH$_{10}$, C$_2$AH$_8$, C$_3$AH$_6$ and γAH$_3$ are the principal hydration product of W.H.A.C. between 16-63°C as shown in fig: II-2.
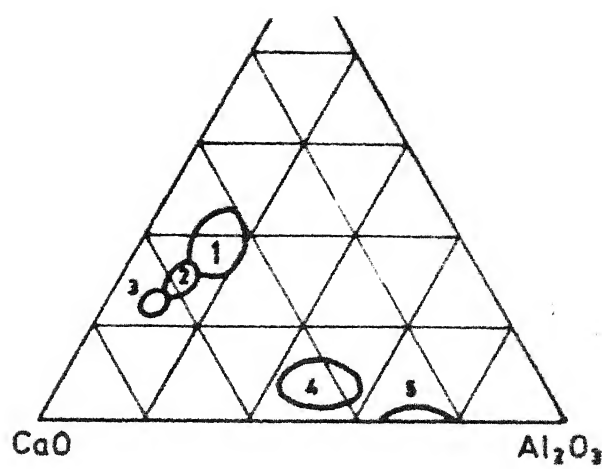
Fig. II-1 The position of the cements in the
CaO – SiO$_2$ – Al$_2$O$_3$ system

  1 Blast-furnace slag; 2 Slag cements;
  3 Portland cements ; 4 Aluminous cement ;
  5 High alumina cement.
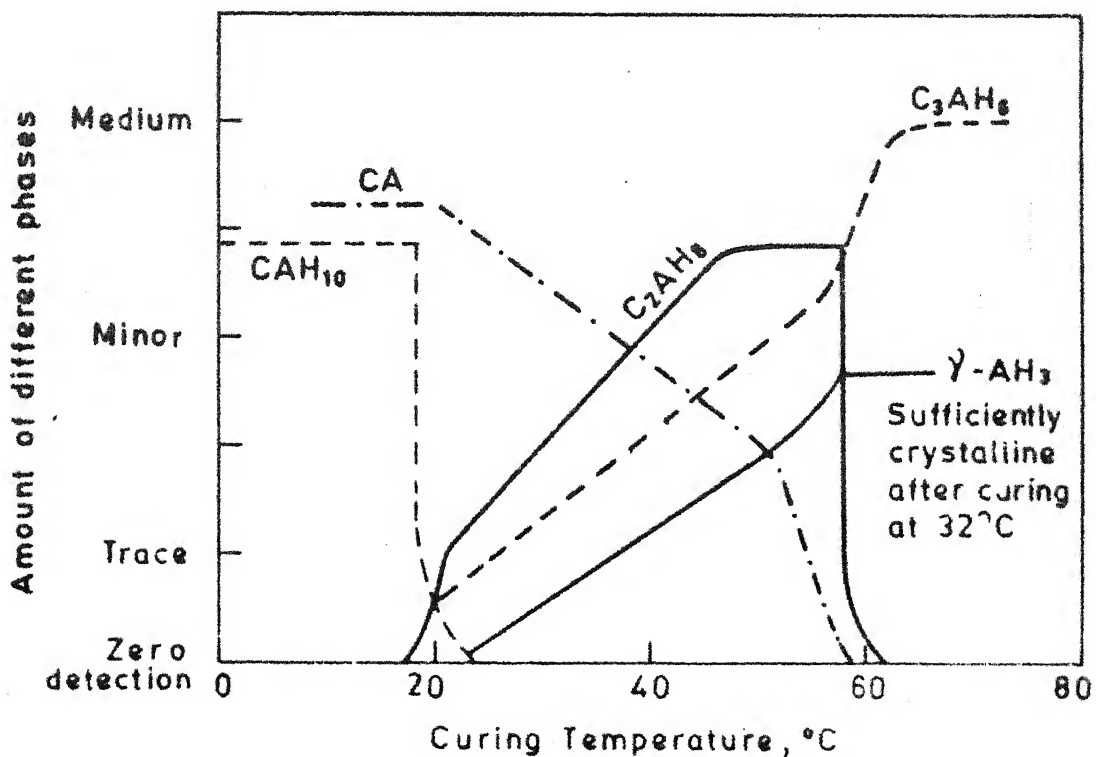
Fig. II-2 Hydration of W.H.A.C. between 16 to 63°C

The hydration reaction scheme is,

$$CA \xrightarrow{<21^\circ C} CAH_{10}$$

$$27-54^\circ C \qquad 27-54^\circ C$$

$$C_2AH_8 + AH_3$$

$$>63^\circ C.$$

$$C_3AH_6 + AH_3$$

$$CA_2 \longrightarrow CAH_{10} + AH_3$$

The overall reaction is,

$$3CAH_{10} \longrightarrow 3/2\ C_2AH_8 + 3/2\ AH_3 + 27/2H \qquad (22-54^\circ C)$$

$$3/2\ C_2AH_8 \longrightarrow C_3AH_6 + 1/2\ AH_3 + 9/2H \qquad (54-63^\circ C)$$

$$3\ CAH_{10} \longrightarrow C_3AH_6 + 2AH_3 + 18H.$$
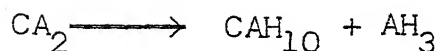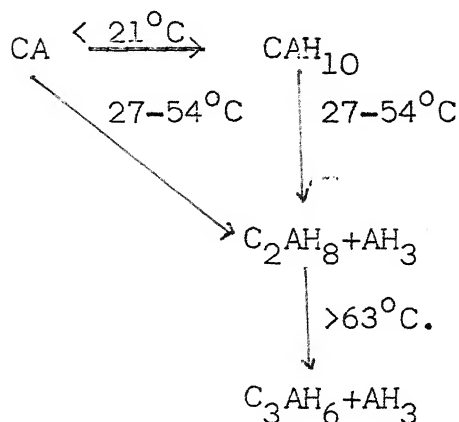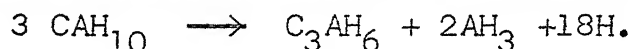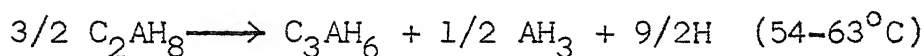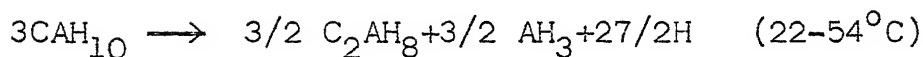
Among the hydration products, CAH_{10} and C_2AH_8 are hexagonal in structure and C_3AH_6 is cubic. Though it is known that complete conversion occurs only above $63^\circ C$, it starts forming at lower temperatures under certain conditions of storage of matured cement specimen. The transformation to C_3AH_6 phase causes loss of strength due to :

i) Conversion of AL_2O_3 gel to micro — crystalline gibsite.

ii)   Formation of lower density products with volume expansion and increase in porosity.

iii) Stresses generated by large size crystals.

C.   PROPERTIES OF W.H.A.C. [7,14].

The specific surface area of W.H.A.C. ranges from about 2500-4000 cm^2/g, commonly about 3000 cm^2/g.

In the absence of ferrites the specific gravity of this cement is around 3.

The cement contains no free lime and sulphur compound and hence is free from any unsoundness.

The setting time of W.H.A.C. is similar to that of portland cement. With 22% water, the typical initial setting time is 2-6 hr. (mean 4 hr.) and final setting time is about 30-45 minutes after the initial set.

Temperature has considerable effect on the setting times as shown in fig: II-3. Setting is fast between 1-18°C, becomes progressively slow from 18-30°C and then again becomes faster above 30°C.

It is note worthy that high alumina cement combines with more water for setting and hidration than
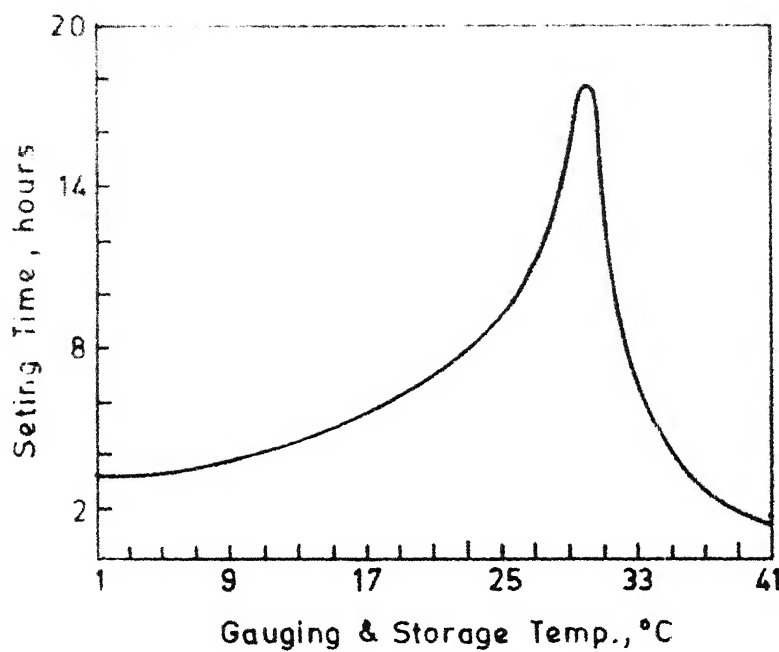
Fig. II-3 Effect of temperature on setting time
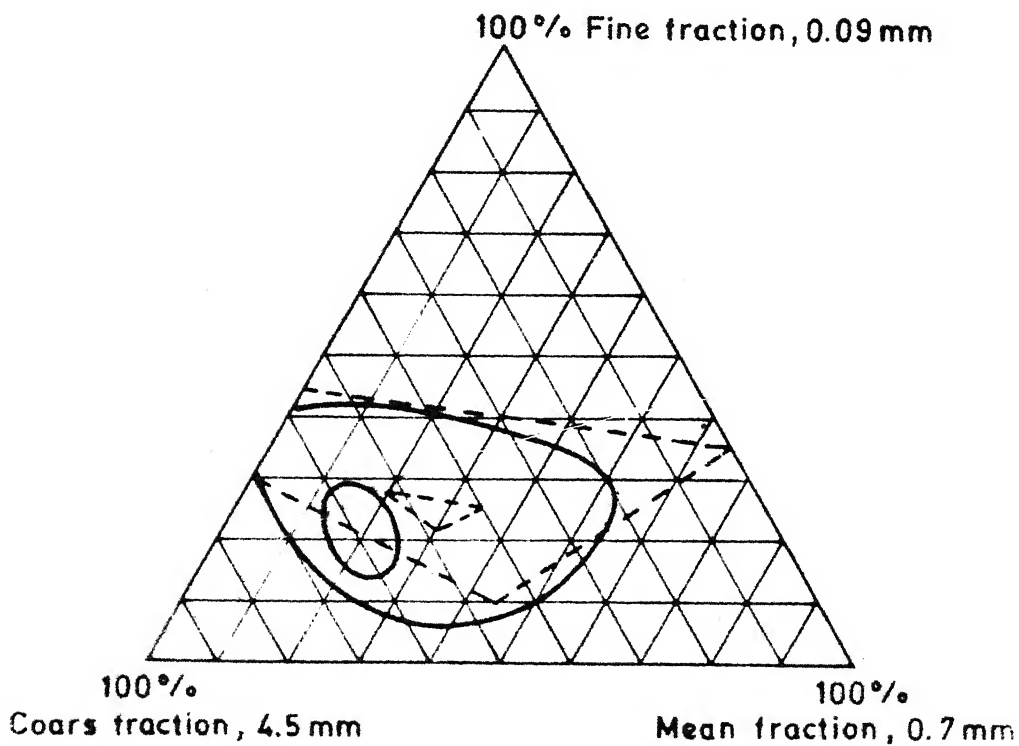
Fig. II-4 Compactness diagram of chamotte: Broken
line shows theoretical compactness; solid
line shows experimentally obtained compactness.

does portland cement. If the initial product is a mixture of CAH_{10} and C_2AH_8 then the water required is as high as 70%; if it is C_2AH_8 and AH_3 then about 50% water is needed; and for C_3AH_6 and AH_3 phases it is about 35%. In nearly completly hydrated set cement, wherein the aluminate has been converted to cubic C_3AH_6 by treatment at $100^{o}C$, the water content is approximately 35% . At ordinary temperature after complete hydration has occured the combined water requirement is about 50%.

At normal temperature high alumina cement attains in 24 hrs. a strength close to its maximum possible. But if the temperature during storage is above the normal, phase conversion takes place and causes loss in strength.

W.H.A.C. differs from portland cement in a number of ways: It is a refractory material, infact some of these are produced by melting rather than sintering; it attains high strength in a few hours rather than few weeks; it exhibit high early heat of hydration; and it gives no free lime on hydration and as a consequence unlike portland cement, it does not undergo cracking due to $CaO \rightleftharpoons Ca\ (OH)_2$ recycling.

3. CONCRETE MIX DESIGN:

A. AGGREGATES:

The chemical nature of aggregates used play a principal role in determining the resistance of concrete to

high temperatures.   The grain size and the grading of the
aggregates are of major importance in determining the
quality of refractory concrete.

By use of suitable graduation of the aggretate
sizes it is possible to achieve a dense packing which in
turn ensures a well consolidated concrete of low porosity
and high structural strength.   The high temperature concerete
looses its hydraulic bond at around 400°C and ceramic bond
begins to form at about 1000°C. As a result cement bonded
concrete passes through a region of minimum compressive
strength,and the packing of aggregates play an important
role in this critical region.

In general the maximum permissible size of aggre-
gate depends upon the minimum size of the member to be const-
ructed.   In general the largest size of aggregate should be
between 1/4 to 1/5th of the minimum thickness of specimen [1].

A. Hummel [15] distinguishes the following types
of grading :

i) Continuous grading, or continuous stages of grain sizes;
the sieve curve of this grading is continuous.

ii) Regulated gap grading, where certain sizes of particles
are delebarately excluded, so as to improve the denseness
of the concrete.   The sieve curve of this grading is

discontinuous in a regular fashion .

iii) Unregulated gap grading in which some sizes are ommited arbitrarily. The sieve curve is discontinuous in an irregular fashion.

The gap grading makes most efficient use of the cement bond but its disadvantages could be the poor workability of wet mix.

In gap grading generally three size fraction i.e. coarse, medium and fine are used, the ratio between the mean size of the fraction lies between 6 and 7.5.

Fig: II-4 shows dense packing contours for coarse, medium and fine fraction in an triaxial diagram. Accordingly the grain composition for maximum packing density is : 55-65% coarse, 10-30% medium and 15-30% fine [16]. Hugill and Rees found that a grading of 45% coarse, 10% medium, and 45% fine gives best packing [17]. It should be noted that optimal packing depends not only on size but also on grain shape.

B. CEMENT CONTENT AND WATER/ CEMENT RATIO:

If the aggregates employed are more refractory than the cement, the refractoriness of the concrete increases as the cement content is reduced. But when the refractoriness

of the aggregate is not greatly different from the cement,
then the cement content has a minor influence. This is
illustrated in fig :II-5.

The effect of cement content on refractoriness
and on green and fired strengths of concrete made with higher
refractory aggregates than the cement is shown in fig: II-6[18].

Provided that the the full compaction of the
concrete mix can be achieved during placing, a reduction in
water/cement ratio invariably gives better strength and
improvement in other concrete properties. In this respect
W.H.A.C. concrete, therefore, obays the same general water/
cement ratio 'law' which applies to portland cement concrete.
It should be noted that it is possible to consolidate W.H.A.C.
concrete with lower water/ cement ratio than in the corres-
ponding mixes made with portland cement.

With porous aggregates the fixing of water/
cement ratio is quite difficult. But with non-porous dense
aggregates like fused alumina, the right ratio can be fixed
easily. The optimal water/ cement ratio is around 0.35-0.4 for
vibration compaction, but if higher pressure is used for
compaction, the ratio is reduced depending on the pressure
used.
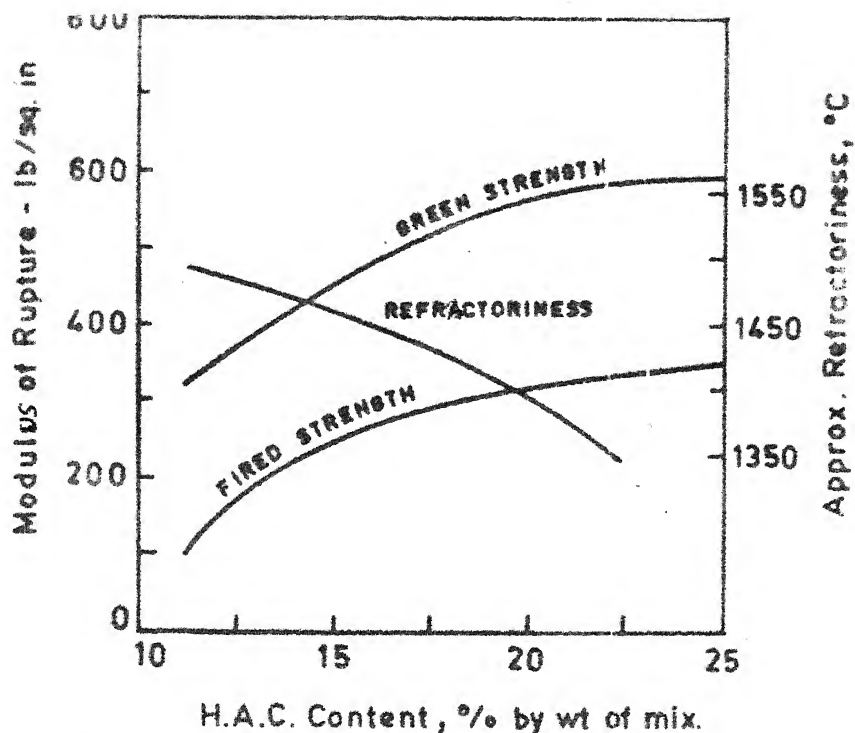
Fig. II-5 Effect of cement content on the refractoriness and strength of castable mix.
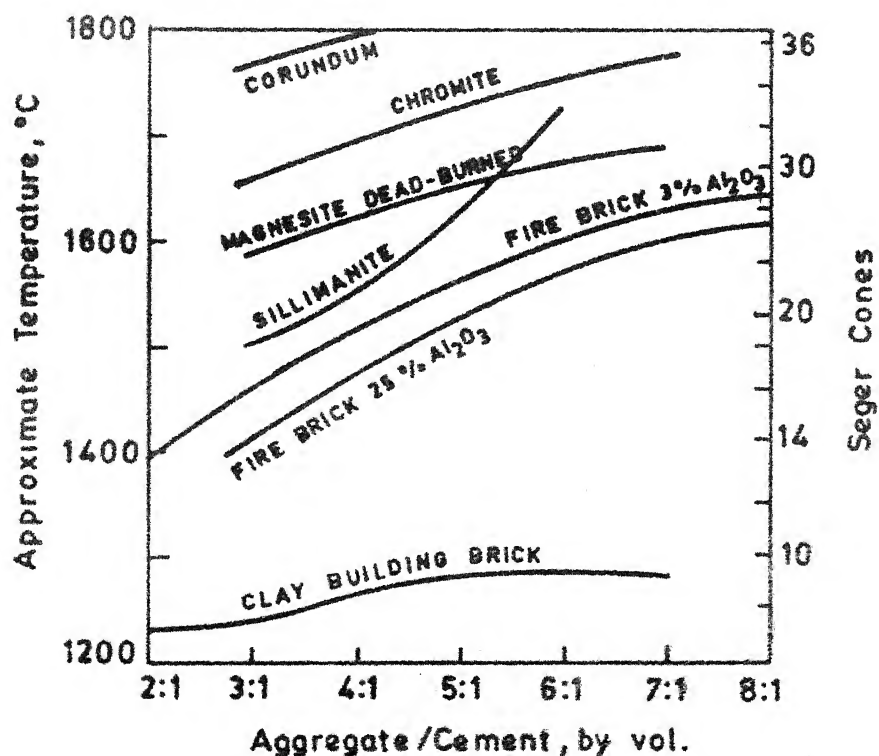
Fig. II-6 Effect of cement content and of type of aggregate on the refractoriness (PCE) of castable mixes.

Mordern developments in process industries such as cement, steel, petrochemicals etc. have imposed severe demands on refractory lining e.g. i) low elastic deformation, ii) high thermal shock and stress resistance,iii) high abrasion resistance, and iv) high alkali resistance. The ramming masses based on tabular alumina bonded with W.H.A.C. can be used successfully to meet these requirements. However these concretes have low permeability due to complex hydrated gel formation. As a result there is a potential danger of powerfull steam explosion if subjected to a fast heating up schedule which is ofcourse needed in these industries in order to save on expensive down time.

This problem has been solved by incorporating upto 0.25 wt% organic fibers which burns out during heat up and allow the steam to escape harmlessly through the channels formed [19].

An alternate solution is to use very low cement amounts, 5-8% and thereby reducing the water requirement. But this procedure leads to a number of problems e.g. limited workability, poor mixing characteristics, difficulties in placing and high sensitivity to water/cement ratio [20]. This problem has been solved by using silica sol containing 40 wt% silica as the

liquid instead of water, Hence cement as low as 2.5% has been used without affecting the unfired as well as fired properties [19].

C. CURING :

After commisioning of the concrete it is necessary to cure it without letting it dry up. Normal procedure is to cure it for 24 hr. in a relative humidity of 90% and then under water. Since concrete with W.H.A.C. hardens very rapidly, evolution of heat due to hydration is quite high. The temperature of concrete may rise excessively leading to a decrease in the strength due to rapid phase conversion . This problem is less acute with thin sections as heat dissipation is easier, but for thick concrete cooling may be necessary. In order to prevent appréciable conve.sion and for producing a normal concrete, it is necessary to ensure that,

i) the maximum temperature of moist concÉrete never exceeds 59°C or so,

ii) the temperature above 38°C is lowered as quickly as possible.

iii) the concrete temperature is below 25°C by the end of 24 hrs. [7].

Although under moist conditions conversion is very slow below 25°C, presence of water is essential for
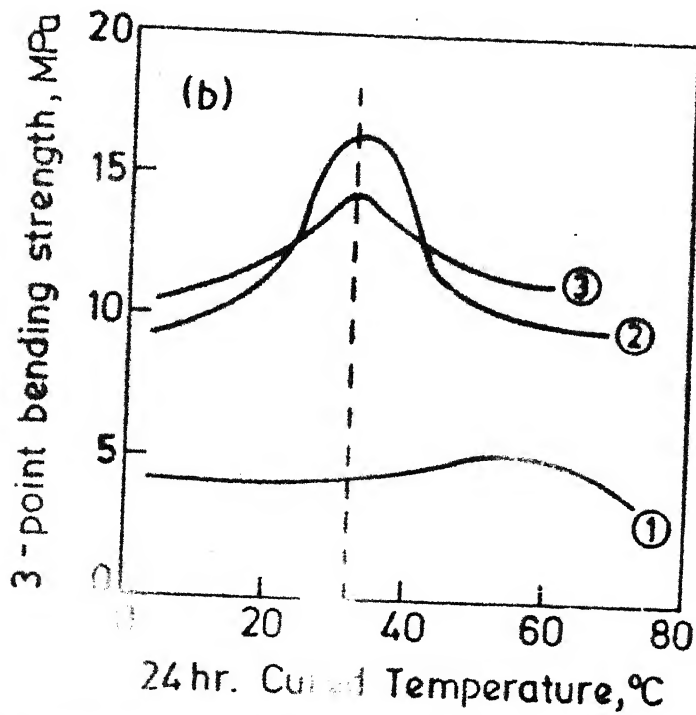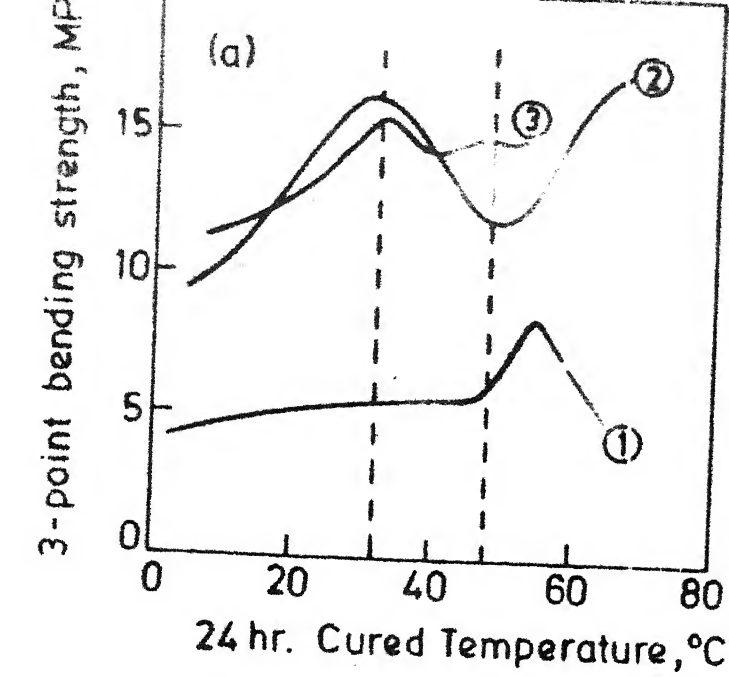
Fig. II-7 Bending strength at different curing temperature
of alumina concrete.
(a) Mixed at room temperature, 22-24°C
(b) Mixed at different curing temperature
1. Cured 24hr ; 2. Dried 110°C, 24 hr ;
3. Dried-fast fired, 1100°C.

conversion at any temperature and once the concrete is dry the conversion does not occur even at high temperatures [14]. Moreover the rate of conversion is less for less water/cement ratio in the concrete mix.

However recently it is reported that a concrete mix of 80% graded tabular alumina and 20% W.H.A.C. has exhibited somewhat anamolous behaviour as shown in fig:II-7. [12,21].

4. HOT PRESSED CONCRETE [22] :

No literature is available on hot pressing [simultaneous heating and pressing] of concrete based on W.H.A.C. and fused alumina aggregates. Very little literature is available on hot pressing of Ca-Aluminate cement only [22,23,24,33,34].

Hot pressing technique was applied to ordinary H.A.C. (40% Al_2O_3) and W.H.A.C. (72% Al_2O_3) at 50,000 psi (3510 Kg/cm^2), $250^{\circ}C$, 1/2 hr and at 50,000 psi (3510 Kg/cm^2), $150^{\circ}C$, 1/2 hr. and the properties were compared with standard paste of the cement . The standard paste was made with w/c = 0.16 whereas the hot pressed body was prepared with w/c =0.8.

Hot pressed cement pastes contained lower percentages of evaporable, non-evaporable and hence total water

as compared to the standard paste. Infact the standard paste contained about six times the evaporable and more than double the total water than in the hot pressed paste.

Hot pressing at $250^{\circ}C$ decreased the porosity, from 22% in the standard paste, to about 4%.

By hot pressing at 50,000 psi and $150^{\circ}C$ the compressive strength of H.A.C. increased by 5 times and at same pressure but $250^{\circ}C$ by about 10 times over that of standard paste. Moreover, the paste hardened and set to almost its full strength, in a very short time of less than 1/2 hr.

Hot pressing of portland cement pastes also gave comparable enhancement in mechanical properties [24].

The reason for this rather dramatic improvement in properties by hot pressing is due to the formation of C_3AH_6 phase directly during pressing itself at higher temperatures employed. C_3AH_6 is the most stable hydrate at elevated pressure in the C-A-H system between room temperature and $300^{\circ}C$ [23]. Hence the potentially destructive phase conversion is thereby avoided . The reaction apparently proceeds as follows,

$$3 [CaO. Al_2O_3] + 8 H_2O \longrightarrow 3CaO.Al_2O_3.6 H_2O+4AlO.OH.$$
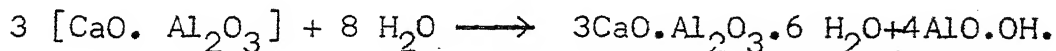
In other words the more stable boehmite is formed rather than gibbsite, $Al(OH)_3$.

In view of above, it is logical to try if strong W.H.A.C. bonded concrete bodies can be formed by hot pressing of the mix, and this was the principle objective of the present work.

5. PHOSPHATE BONDED ALUMINOUS AGGREGATES:

Kingery [28] has stated that phosphote bonding is accomplished by three methods (i) reaction between silicious materials and phosphoric acid, (ii) reactions between oxides and phosphoric acid, and (iii) direct addition or formation of acid phosphates. Besides phosphoric acid other bonding agents like monoaluminioum phosphate, amonium phosphate, chromium- aluminium phosphate etc. are also used.

Phospharic acid reacts with amphoteric oxides fabourable in the sense that the reaction occurs at a controllable rate. But the problem with H_3PO_4 as the binding agent in that the refractory may bloat during heating due to evolution of hydrogen formed by the reaction of the acid with metallic impurities present in the mix. To minimise this problem, monoaluminium phosphate (MAP) is generally used. The phases formed by treating Al_2O_3 with H_3PO_4 or MAP are reported to be same.

The most important characteristics of the aluminium acid phosphate bond is its acidity x defined as the P_2O_5: Al_2O_3 molar ratio. Mixture with $x < 3$ are metastable and

bellow 2.3 are untable. Typical commercial MAP products have x = 3. The phases that are formed by the reaction of H_3PO_4 and Al_2O_3 at various temperatures are described below [29].

Hydrogen bonded polymers are formed at lower temperature range ($500-800^{\circ}C$), and then condense to glassy or amorphous macromolecules at higher temperatures ($1000-1300^{\circ}C$). At still higher temperature, the glassy phase crystallizes and the phosphate decomposes thermally.

The strength and stability of the phosphate bonded alumina bodies can be enhanced through the use of chromic acid as partial replacement of phosphoric acid [30]. Highly colloidal polyphosphates can be produced by incorporating Cr_2O_3 in the binder composition $Al_2O_3 \cdot 3P_2O_5 \cdot x\, Cr_2O_3$, where x may vary from O.1 to 1.O. The tendency of crystallization and volatalization of these polyorthophosphates are considerably less than in the case of MAP. Roy and Sircar[31] have concluded that the hot strength enhancement is due to (1) retention of a portion of P_2O_5 in glassy state either as aluminium-chromium phosphate or chromium phosphate and (ii) reinforcement of partly crystalline aluminium phosphate with either aluminium chromium phosphate glass or chromium phosphate glass.
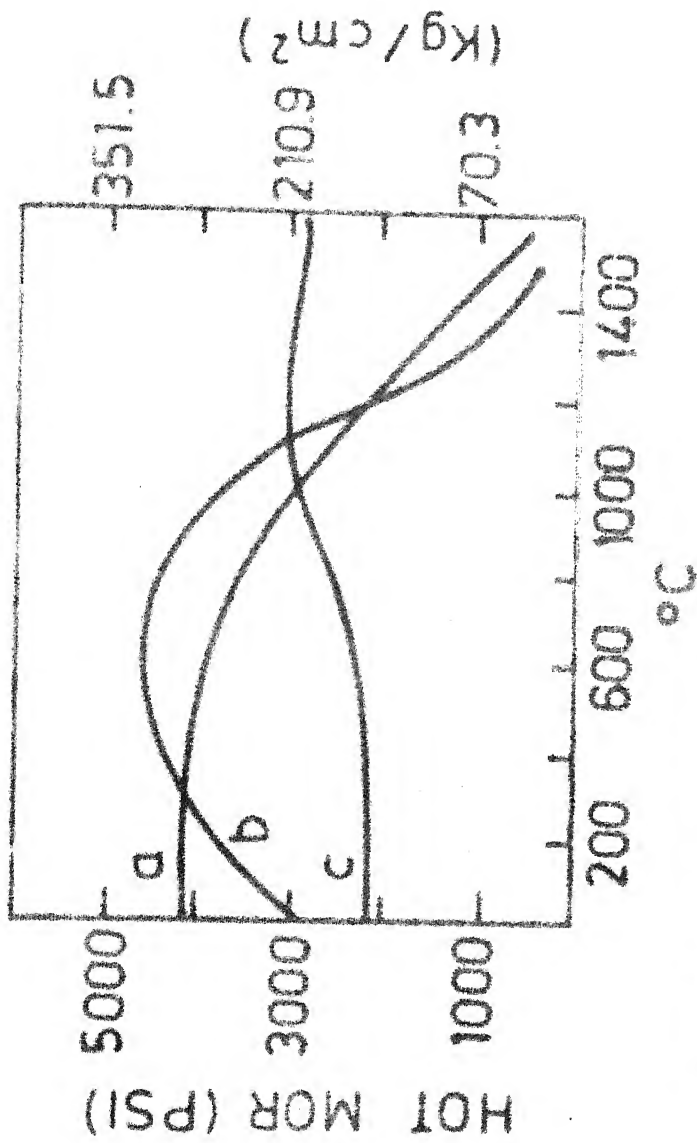
Fig. II-8: Hot MOR of refractory bodies with tabular Al_2O_3 base, fired at 1700°C and bonded with (a) calcined Al_2O_3, (b) calcined $Al_2O_3 \cdot H_3PO_4$ and (c) calcined $Al_2O_3 \cdot SiO_2$.

Phosphate bonded high alumina bricks, morters, ramming mixes, plastic and castables are now widely used. In general these (i) do not show week regions on heating, (ii) have good refractoriness, (iii) posses high abrasion resistance at elevated temperature and (iv) have good slag resistance and melt nonwetting property. Polfreman [32] has described the properties of a veriety of high alumina based pressed bricks bonded with H_3PO_4 and compared them with other kinds of alumina bricks. As shown in fig: II-8, the phosphate bonded product has good strength at modarate temperatures but it drops rapidly at higher temperatures $(> 1000^{o}C)$.

Phosphate bonded high alumina bricks are used in electric arc furnace roof lining and also in lining for blast furnace runners and in laddle. They are also useful for nonferrous applications such as hearth and lower side walls of aluminium re-melting and holding furnaces.

CHAPTER - III

OBJECTIVE OF THE WORK

The objective of this work was to prepare W.H.A.C. bonded fused alumina bodies of high strength. The strength of concrete can be increased by many ways,

1. Grinding the cement finer.
2. Dense packing of aggregates.
3. Decreasing water/ cement ratio.
4. Using high molding pressure.
5. High temperature of pressing.
6. Maintaining the sample under load for longer time.

Initial investigation showed that it was not possible to grind the cement finer in available laboratory ball mill because of excessive agglomeration and coating of the grinding media and the mill by the cement. Lack of time prevented from employing alternate grinding machine such as air jet mill, vibro-mill etc.

All the other above mentioned methods for increasing the strength were incorporated in the present investigation, although not in the same order as described above. In order to find out the extent of improvement possible by

hot pressing, initial trials were done on cold pressing only. Comparison were also made with phosphate bonded alumina to which another set of experiments were conducted.

The core of objective of this investigation was to determine the optimal aggregate mix, cement content, water/ cement ratio and molding pressure with the objective to attain as high strength as possible with :

1. Minimum cement content.
2. Low water/ cement ratio.
3. Low molding pressure.

CHAPTER-IV

MATERIALS AND METHODS

1. MATERIALS:

   A. ALUMINA:

   i) Brown Alumina :

   This alumina is produced directly from bauxite by electrofusion. The brown color is due to Fe_2O_3 and TiO_2 impurities . Its specific gravity is about 4. The chemical analysis of brown alumina used in these experiments, as stated by the suppliers [CUMMI] was ,

TiO_2	2.25-2.65%
SiO_2	0.3-0.9%
CaO	0.6-0.8%
Fe_2O_3	0.2%
Al_2O_3	96%

The gradings of this alumina was ,

Grit No.	10	24	100	200
Average size, mm.	1.651	0.701	0.15	0.075

ii) White Alumina:

White alumina is produced by electro-fusing commercial Bayer's alumina . Its melting point is higher than that of brown alumina in view of the lesser amount of impurities. The chemical analysis was, 99.5% Al_2O_3 with 0.33% Na_2O and < 0.1% SiO_2 and Fe_2O_3 each. The grading used was-200 grit (-0.075 mm).

iii) Bayer's Alumina:

This alumina is made from bauxite by Bayer's process. This alumina contained, 0.35-0.7% Na_2O; 0.01-0.1% CaO; and 98.5% Al_2O_3 with tracess of Fe_2O_3 and TiO_2.

Compositions of aggregates used:

	Grits of fused alumina, wt%				Bayer's alumina, 72 hrs.ground, wt%
	10	24	100	-200	
Batch-1	50	15	15	20-WHAC	—
Batch-2	50	15	15	10	—
Batch-3	50	25	10	6	9
Batch-4	50	10	10	20	10

B. CEMENT:

The cement used was CAL-AL-75 grade high purity white high alumina cement (W.H.A.C.) from ACC. This cement is made by clinkering or fusing together pure grades of alumina and calcium carbonate. The chemical analysis and specifications of the W.H.A.C. used in this study is given below.

Chemical composition:

Al_2O_3	73.5%
CaO	23.6%
SiO_2	0.7%
Fe_2O_3	0.3%
MgO	0.8%
Sp.surface area	3500 cm^2/gm
Sp.gravity	2.9-2.95
P.C.E.	1690-1710°C
Phases	CA and CA_2 Major
	$C_{12} A_7$ and A Minor

Setting time

Initial	60 min. (minimum)
Final	600 min. (maximum)

Compressive strength

1:3 mortor cube

250 Kg/cm^2(6 days)

450 Kg/cm^2(dried, 110°C)

C. <u>CHEMICALS</u>:

i) Ortho phosphoric acid $[88-93\%]H_3PO_4$ of molecular wt. 98.00 and density at $20^{\circ}C$: 1.75 gm/cm^3 was used.

ii) Chromium trioxide (CrO_3), LR grade of mol. wt. 100.01 was used.

2. <u>METHODS</u>!

A. <u>GRINDING</u>:

Grinding was done in ceramic jar of height 21 cm and internal diameter 28.5 cm. The grinding media was alumina cylinders of diameter 2.065 cm. and height 2.072 cm.

Bayer's alumina was ground wet and the weight ratio of alumina: water : grinding media was 1:1:3. The volume of slurry taken filled 60% of the jar volume with grinding media. Grinding time was 72 hrs. The r.p.m. of the jar was 60, The ground alumina was washed out from the mill and oven dried.

B. <u>PREPRATION OF PHOSPHATE BOND</u>:

The bond was prepared by mixing weighed amount of H_3PO_4 and CrO_3 to get the desired molar ratio of P_2O_5: Cr_2O_3 of 1:4 and heating the mixture to $100^{\circ}C$ to get a almost homogeneous solution.

C. PREPARATION OF TEST SAMPLES:

i) Cement Bonded:

a) Mixing :

Weighed amounts of different gradings of alumina and cement were hand mixed dry. A known volum of water was added and quickly mixed with hand as thoroughly as possible. The time of mixing with water was 2 to 3 minutes.

b) Cold and Hot Pressing:

The moist mix was pressed in a steel die in a hydraulic 50 tonnes press.

Hot pressing was done by winding a resistance coil around the die and connecting it through a variac. The temperature was adjusted by adjusting the voltage in the variac. The procedure was as follows, the main cylinderical part of the die was placed on the die base and the pressing punch was inserted. The die was heated up until the temperature just exceeded $100^{o}C$ as measured by a thermometer inserted in the die after the withdrawal of the top punch. The variac was adjusted to maintain this temperature. Water was added to the drymix and the wet mixture at ambient temperature of $20-30^{o}C$ was introduced in the die, pressed down with the punch and immediately loaded in the

hydraulic press to the total required load. It should be noted that the total die mass was 5.8 Kg and the concrete sample weighed about 100 gms. Therefore due to the high thermal mass of the die, the temperature inside the die was expected to drop only marginally bellow 100°C. This was confirmed by the observation in the initial experiments when steam use to escape from between the die base and the cylinder joints, leading to loss of moisture and drying up of the sample at the lower portion. To prevent that thick polythin sheet was cut and placed on the die base plate and it acted as a gasket.

The pressed sample was stripped out of the mold carefully.

The sample diameter was 5 cm. and thickness was about 2 cm.

c) Curing :

The pressed samples were kept in about 90% relative humidity for 24 hr. in a descicator filled with water at the bottom. The descicator was kept inside an oven maintained at 30-35°C. The sample were taken out of the desciator after 24 hrs. and kept under water for 7 days at 30-35°C inside an oven.

The cured samples were dried in an air oven for 24 hrs. at temperature of 100-110°C.

ii) Phosphate bonded:

a) Mixing:

Weighed amount of graded alumina were taken in a morter-pestle and known volume of phosphate bond was added and mixed throughly for 10 minutes. The preground Bayer's alumina was used as the bonding matrix.

b) Pressing:

Green mix was pressed under a hydraulic press. The pressed samples were stripped out carefully.

Hand rammed cubes were also made in a standard cemer testing mold.

The pressed/ molded samples were dried in an air oven for 24 hr. at a temperature of 100-110°C.

c) Curing:

Curing was done in an electrically heated autometically controlled furnace at different temperatures e.g. 400°C, 600°C and 800°C. The time of soaking for each temperature was 5 hrs. The rate of heating was as follows,

Room temperature to $400^{\circ}C$ - 2 hrs.

$\quad$ 400 to $600^{\circ}C$ $\qquad$ - $2\frac{1}{2}$ hrs.

$\quad$ 600 to $800^{\circ}C$ $\qquad$ - 3 hrs.

After curing the samples were cooled overnight inside the furnace .

D. **TESTING OF THE SAMPLES :**

i) Bulk Density:

The thickness [t, in cm.] and the diameter [D, in cm.] were measured to calculate the volume and weight was taken [W in gms].

The bulk density was computed by using the following relationship.

$$\text{Bulk density} = \frac{4\ W}{\pi D^2 .\ t} \quad \text{gms / cm}^3.$$

ii) Splitting Test (Brazilian Test) :

The cylinderical samples were placed in such that their axes were horizontal to the platens of the testing machine and the compression load was exerted on the curved surface. The failure occurs by splitting the sample along the vertical diameter.

The instrument used was INSTRON 1195 model. The loading parameters were,

Crosshead speed   :   0.5 mm/min.

Chart speed   :   20 mm/min.

The final load to fracture P is related to the tensile strength by following:

$$\text{Tensile strength} = \frac{2P}{\pi.t.D} \quad Kg/cm^2.$$

Where,

P: Load to fracture [Kg]

t: Thickness of the sample [cm]

D: Diameter of the sample [cm]

CHAPTER -V

RESULTS AND DISCUSSIONS

1. COLD PRESSED CONCRETE.

In order to acquire base data for comparision with hot pressed samples, first cold pressing of concrete was carried out with different process parameters and the samples were tested for bulk density and tensile strength.

A. EFFECT OF CEMENT CONTENT:

Fused alumina aggregates of composition batch-1 were pressed with varying cement contents and pressed into 5 c.m. diameter discs under 10 and 20 tonnes total load.

From Fig. V-1 and table : A-1 in appendix , it is seen that at 20 tonnes total pressing load, the bulk density increases gradually as the cement content increases from 5 to 20% in the range of 2.95 gms/cm^3 to 3.16 gms/cm^3. At 10 tonnes pressing load the b.d. at first drops as the cement content is increased from 5 to 7.5% . After that it also increases gradually with the increase in cement content upto 20%, in the range of 2.92 gms/cm^3 to 3.05 gms/cm^3. The mean difference in bulk densities at these two moulding pressures was of the order of 2.3%.
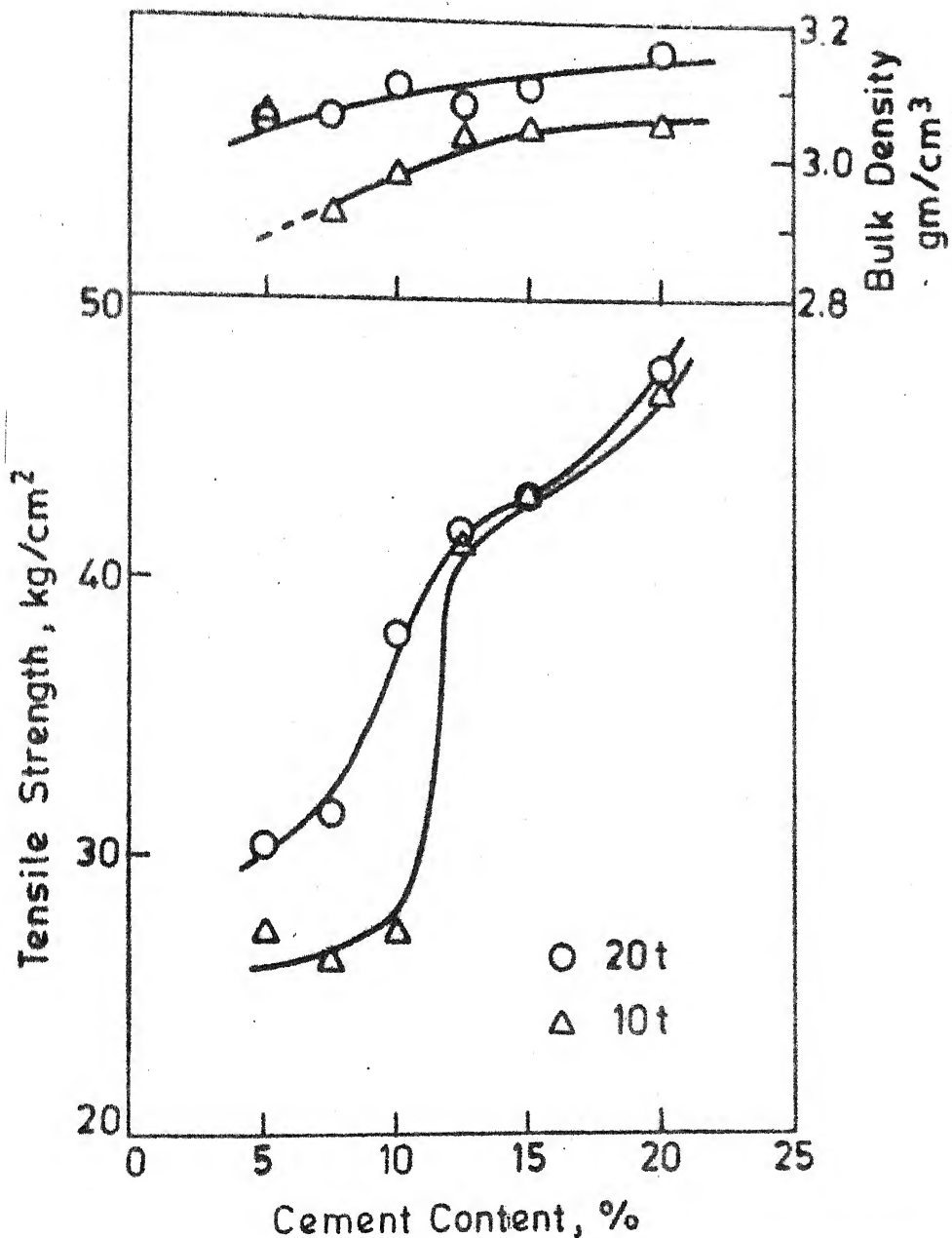
Fig. V-1 Effect of cement content on bulk density and tensile strength of cold pressed concrete.

COMPOSITION: Batch - 1
PRESSING LOAD: 20t and 10t
WATER/CEMENT RATIO: 0.33 for 20 t
0.36 for 10 t

SAMPLE DIAMETER: 5 cm
CURED: 30-35°C in 90% RH, 24 hr
30-35°C under water, 7 days
DRIED: 110°, 24 hr

From the fig. V-1 and table : A-1 it is seen
that in general the tensile strength increases with increa-
sing percentage of cement content. At both the pressures
the increase in t.s. is rapid in the range of 7.5 to 12.5%
cement and this is even more pronounced for 10 tonnes
pressing load. Above 12.5% cement content the rate of
increase of t.s. becomes negligible.

At low cement contents there is marked difference
in the t.s. values for 20 and 10 t. pressing load. But
as the cement content exceeds 10%, the difference begins
to narrow down and eventually the effect of the molding
pressure almost vanishes beyond 12.5% cement i.e. the
strength values almost coinsides with each other. In other
words one can use 10 tonnes load rather than 20 tonnes if
the cement content is more than 12.5%.

B. EFFECT OF WATER/ CEMENT RATIO:

It is weell known that water/ cement (w/c) ratio
has a profound effect on the strength of concrete. This is
because the water needed for hydration of cement is invari-
ably much less than the actual water used, otherwise the
mix does not have enough workability. As a consequence
the excess water, over and above that needed for hydration,
also called evaporable water, leaves behind pores on drying
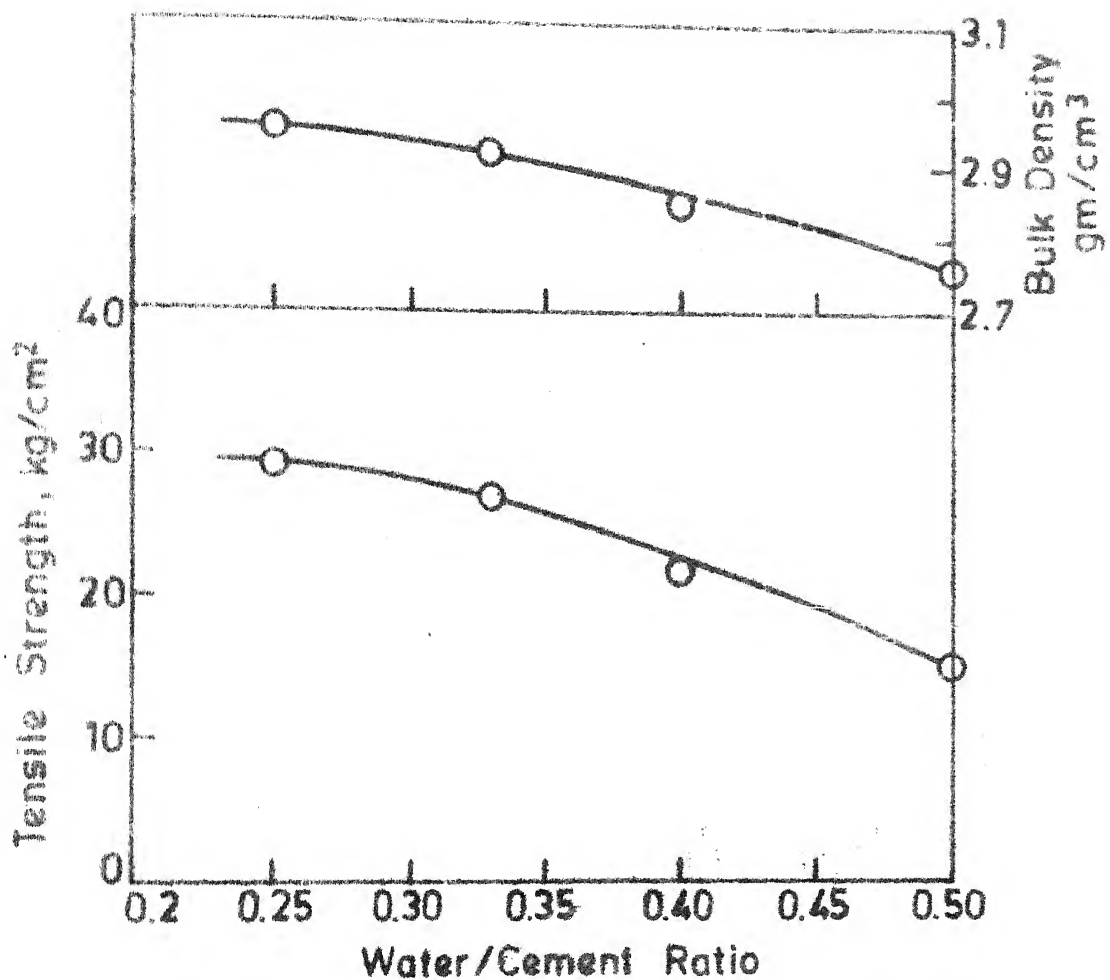
Fig. V-2 Effect of water/cement ratio on bulk density
and tensile strength of cold pressed concrete.

COMPOSITION : Batch-2
CEMENT CONTENT : 10%
PRESSING LOAD : 10 Tonnes
SAMPLE DIAMETER : 5 cm
CURED : 30-35°C in 90% RH , 24hr
         30-35°C under water , 7days
DRIED : 110°C, 24 hr

which naturally reduces the strength. Clearly high molding pressure can tolarate less water than hand molding. In this part of the study the effect of w/c ratio on the t.s. of molded pieces was investigated . The composition was batch-2 with 10% W.H.A.C. pressed under total load of 10 tonnes, the piece again had 5 cm. diameter. Details are given in table: A-2.

From fig. V-2 it is seen that as the water/ cement ratio decreases both bulk density and tensile strength increases. In the range of 0.5 to 0.33 w/c ratio the rate of increment is faster but bellow 0.33 the rate becomes quite slow.

It should be noted that when w/c ratio used is less than 0.33, the handling of given samples at least at

10 tonnes pressing load becomes

difficult, because of its dry structure and friable edges. Therefore a w/c ratio of 0.33 is chosen for further studies.

C. EFFECT OF MOLDING PRESSURE:

Using the same composition as before i.e. batch-2 with 10% cement and w/c ratio 0.33, the effect of molding pressure on b.d. and t.s. was investigated. The data is given in table : A-3 in appendix.
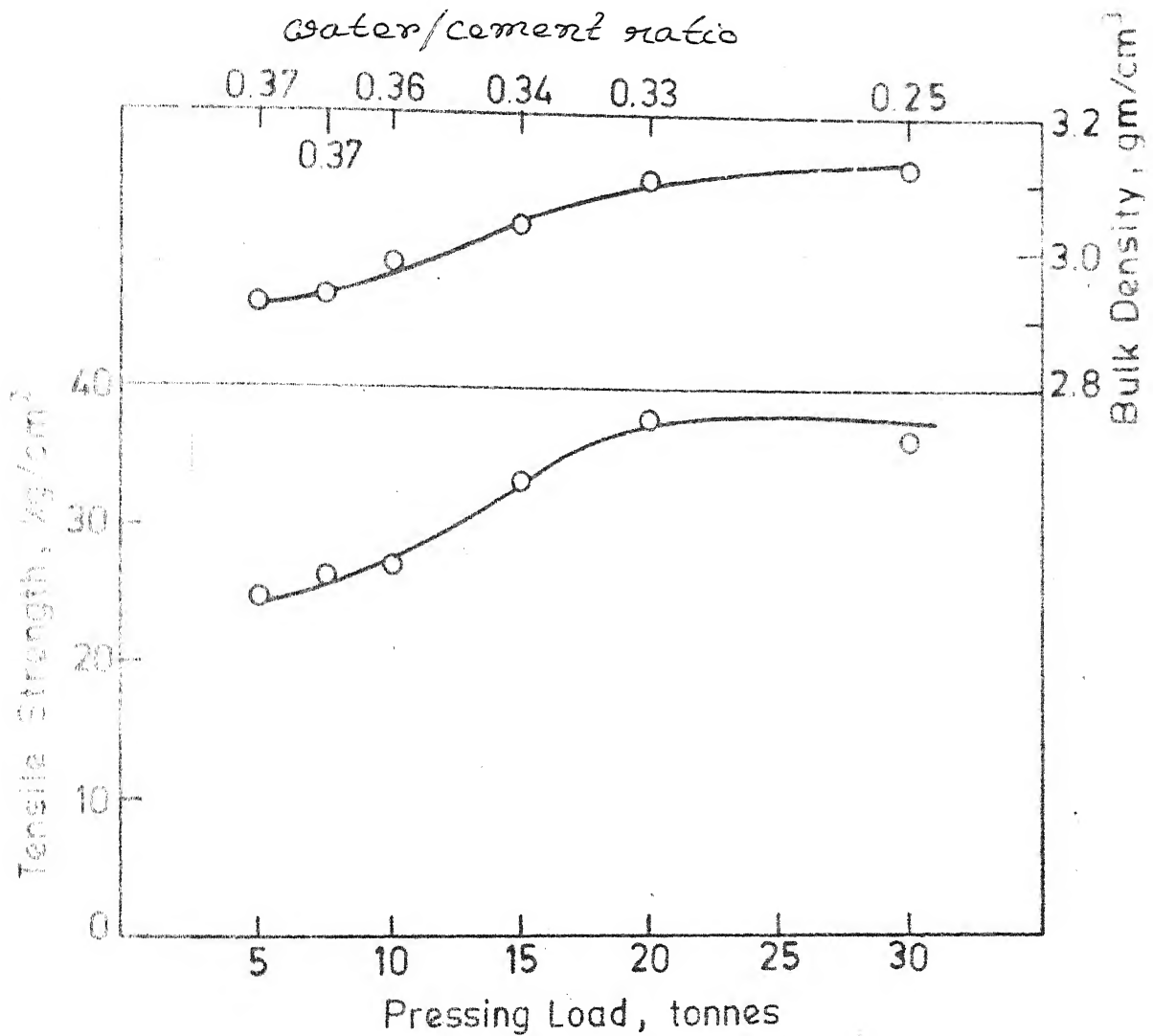
Fig. V-3 Effect of pressing load on b.d. and t.s. of cold pressed concrte

Composition : Batch - 2
Cement content : 10%
Cured : 30 - 35°C in 90% RH, 20 hrs
          30 - 35°C under water, 7 days
Dried : 110°C, 24 hr

It is seen in fig. V-3 that with increasing molding pressure i.e. pressing load, the b.d. and t.s. follow nearly parallel path. Upto 10 tonnes pressing load b.d. and the corresponding t.s. increases slowly. Between 10 and 20 tonnes the increase in much steeper and between 20 to 30 tonnes the values almost flatten out. It would be seen that for maximum density and strength 20 tonnes load is adequate. Hence this was the maximum pressure used in hot pressing.

2. HOT PRESSED CONCRETE:

A. OPTIMAL PROCESS CONDITIONS FOR HOT PRESSING:

As pointed out in the literature survey, strength of set cement can be increased significantly if the wet mix is pressed at elevated temperature. For this part of the study, due to experimental limitations, the temperature of pressing was fixed at about $100^{\circ}C$ although much better results could have been obtained at still higher temperature. The time under full load during pressing was fixed at 10 minutes, again better results could have been obtained with longer holding time.

The composition chosen for this study was batch-**1**. The cement content ranged from 7.5 to 20%. The water/cement

ratio varied from 0.33 to 0.1. The minimum pressing load was 5 tonnes and the maximum was 20 tonnes.

In order to locate the optimal process conditions within the range of process variables mentioned above, normally it would have been necessary to carry out a very large number of experiments. To avoid this labour a Monte-Carlo based random search was conducted. In this search the levels of process variables are,

Cement conten : 7.5, 10, 12.5, 15 and 20%
Water/ cement ratio: 0.33,0.2, 0.14, 0.13,0.11 and 0.1
Pressing load : 5,7.5, 10, 15, 20 tonnes.

These levels were noted on pieces of papers, these papers were folded and well mixed, and randomly drawn with replace-ment . This procedure was repeated for each of the three process variable to give a random set of compositions for conducting the experiment. Table : V-1 gives 19 such compositions selected by this search. Fig.:V-4 shows in bar diagrams the b.d. and t.s. of each compositions. Table:V-1 gives the numerical datas.

Some general observation can be made by detailed scrutiny of fig : V-4. Other things being equal,

TABLE : V-1

Monte-Carlo search for optimal cement content, water/cement ratio and molding pressure in hot pressing.

Composition :

Batch – 2	Grit no.	wt%
Fused Alumina	10	50
	24	15
	100	15
	-200	20 – W.H.A.C.
W.H.A.C.		Rest

Sample diameter	:	5 cm.
Cured	:	30-35°C in 90% r.h., , 24 hr.
	:	30-35°C under water, 7 days
Dried	:	110°C, 24 hr.

No.	Cement Content %	Water/ Cement Ratio	Pressing Load , tonnes	B.D. [gm/cm^3]	T.S. [Kg/cm^2]
1.	17.5	0.14	5	2.828 2.860	32.06 35.14
2.	20	0.2	7.5	2.921 2.901	48.31 47.92
3.	7.5	0.11	1.5	–	–
4.	7.5	0.33	5	–	–
5.	12.5	0.14	20	3.026 2.907	50.34 50.68
6.	7.5	0.2	15	–	–
7.	10	0.1	10	2.814 2.922	25.60 27.81
8.	7.5	0.14	7.5	–	–
9.	20	0.11	20	3.082 3.085	73.20 73.69
10.	12.5	0.14	10	2.959 2.928	49.08 50.02
11.	15	0.13	7.5	2.890 2.893	37.93 38.35
12.	15	0.33	10	2.956 2.984	47.54 49.53
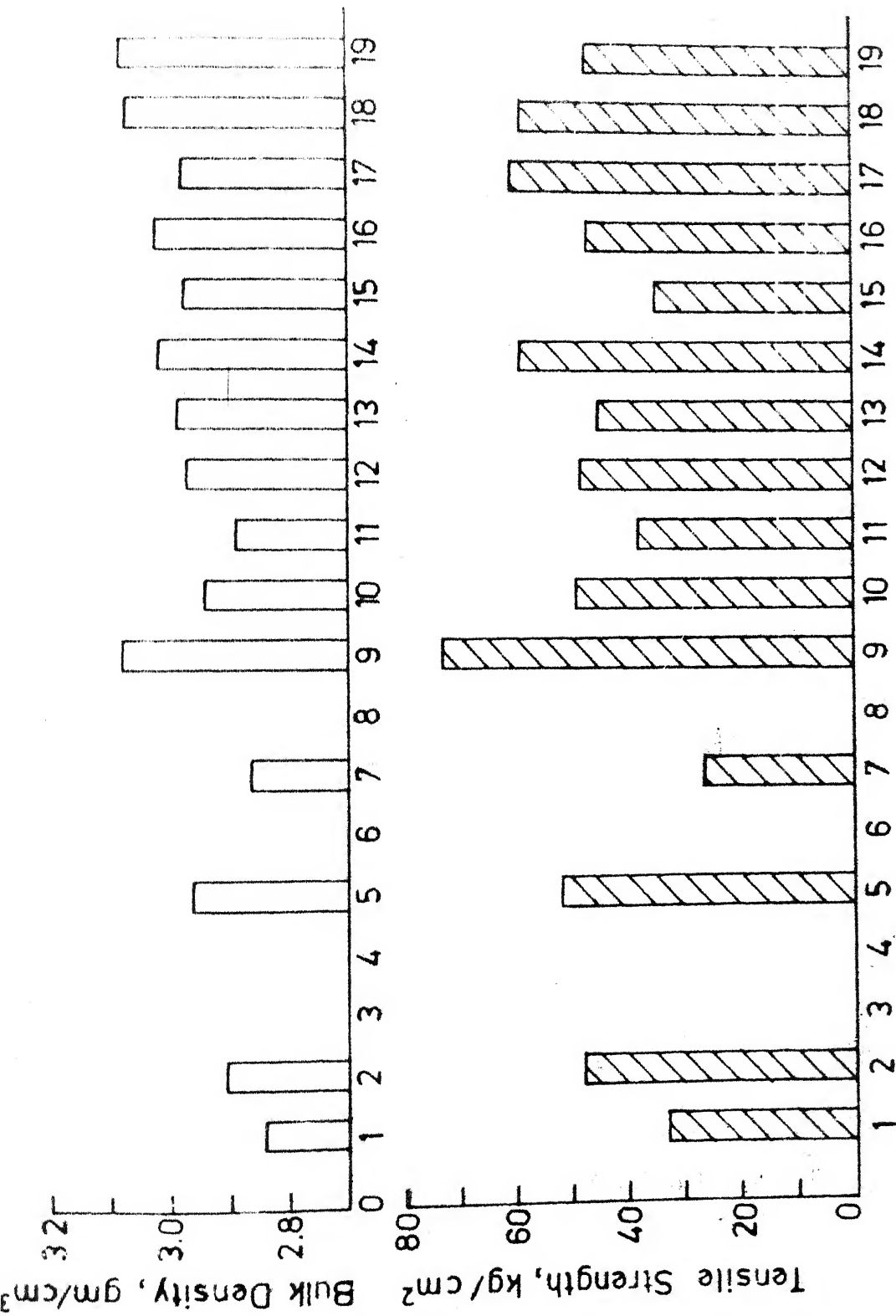
Fig. V-4 Monte-Carlo search for optimal cement content, water/cement ratio and pressing load in hot pressing.

Composition: Batch-2, Sample diameter: 5cm, Cured: 30-35°C in 90% RH, 24hrs
30-35°C under water, 7 days
Dried: 110°C, 24hrs

i) both t.s. and b.d. increase with increase in cement content.

ii) both b.d. and t.s. increases with decrease in w/c ratio.

iii) both increases with increase in pressing load.

iv) in all the cases the t.s. is significantly higher than the cold pressed concrete having similar composition, cement content and pressing load.

The maximum t.s. obtained was of the order of 75 Kg/cm^2 using 20% cement, 0.11 w/c ratio and pressing load of 20 tonnes.

The superiority of hot pressing can be attributed to a number of factors,

i) at high temperature the viscosity of water drops. Infact the water in the die is partly in vapour phase, this permits a much lower w/c ratio than in the cold pressing.

ii) the rate of reaction is accelerated due to higher temperature. Since the cementing reaction is taking place under heavey load i.e. under very good particle-particle contact a strong cementing bond is formed.

iii) the stable cubic hydrate C_3AH_6 in the C-A-H system of the cement phase is formed directly instead of unstable hexagonal hydrates like CAH_{10} and C_2AH_8. Also

does not exceed 80. Then d was picked and checked that, (a + b + d) does not exceed 100. Next c was computed by using constraint no. (iii) Finally the amount of -200 grit alumina was computed from d negetive the cement content.

For each compositions in the previous series 10 compositions of grading were randomly chosen for further tests.

The results are discussed below :

For the improvement in composition no.5 the data for 10 compositions are shown in table:V-2 and the results are exhibited in fig : V-5 . It would be seen that 5_2 and 5_3 are the best attainable results with a marginal improvement.

For composition no.9 the data is shown in table: V-3 and the results are exhibited in fig: V-6. ompositions 9_1, 9_3 and 9_5 seems more promissing, each exceeds 70 Kg/cm^2 tensile strength.

For composition no. 17 the data is given in table: V-4 and the results are exhibited in fig: V-7. Compositions 17_2 and 17_9 gave promising results. In the former case the strength went up by 5 Kg/cm^2.

The fact that Monte-Carlo search on these 3 compositions does not lead to any dramatic improvement is

TABLE : V-2

Monte-Carlo search for obtimal grading of aggregates with comp. no. 5 in hot pressing.

Cement Content	:	12.5
Water/Cement Ratio	:	0.14
Pressing Load	:	20 t.
Sample Diameter	:	5 cm.
Cured	:	30-35°C in 90% r.h., 24 hr.
	:	30-35°C under water, 7 days
Dried	:	110°C, 24 hr.

No.	grit no. wt%				B.D. $[\text{gms/cm}^3]$	T.S. $[\text{Kg/cm}^2]$
	10	24	100	-200		
1.	70	0	0	30	2.959	40.79
					2.936	36.18
2.	50	10	10	30	3.026	55.32
					3.088	56.08
3.	50	10	0	40	2.986	50.91
					3.001	55.98
4.	60	10	10	20	3.027	48.22
					3.011	45.33
5,	50	0	10	40	2.952	49.42
					2.981	49,91
6.	40	20	10	30	2.966	40.12
					2.932	39.85
7.	40	0	20	40	2.954	35.62
					2.963	37.28
8.	60	0	10	30	2.907	41.41
					2.915	41.63
9.	60	10	10	20	2.981	46.28
					2.970	44.32
10.	70	10	0	20	2.933	43.39
					2.912	45.42
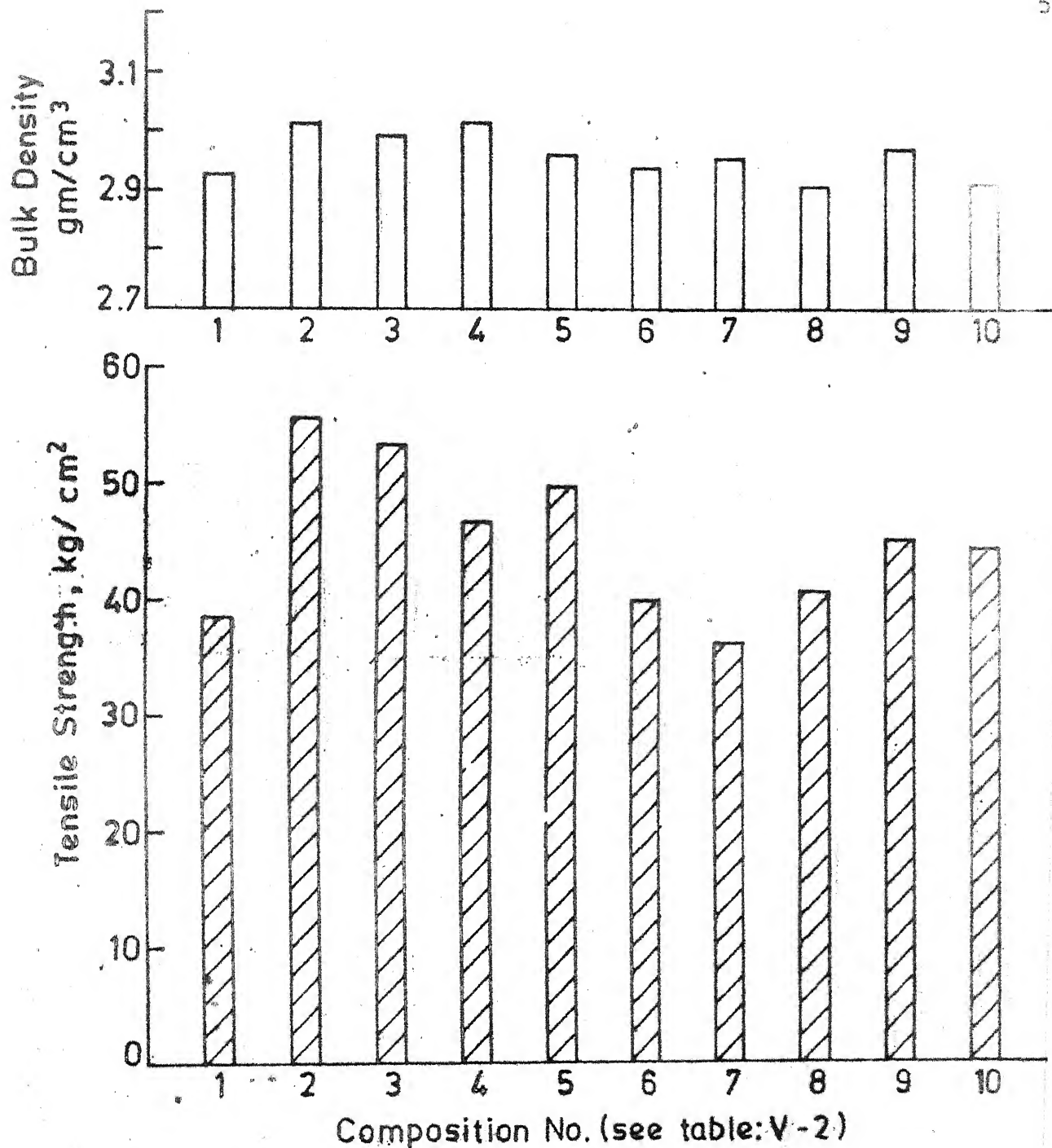
Fig. V-5 Monte-Carlo search for optimal grading of aggrega es in composition No. 5.

TABLE : V-3

Monte-Carlo search for optimal grading of aggregates
with composition no. 9 in hot pressing .

Cement content	:	20%
Water/ Cement ratio	:	0.11
Pressing load	:	20 tonnes
Sample diameter	:	5 cm.
Cured	:	30-35°C in 90% r.h., 24 hr.
	:	30-35°C under water, 7 days
Dried	:	110°C, 24 hr.

No.	grit no.wt%				B.D. $[\text{gms/cm}^3]$	T.S. $[\text{Kg/cm}^2]$
	10	24	100	-200		
1.	50	10	0	40	3.699	69.58
					2.986	71.69
2.	60	10	10	20	3.013	65.39
					3.033	65.71
3.	50	0	10	40	2.970	70.03
					2.932	-
4.	70	0	10	20	2.949	55.28
					2.983	58.96
5.	40	10	10	40	3.066	76.43
					3.024	70.36
6.	70	10	0	20	3.033	58.49
					3.038	62.02
7.	50	20	0	30	3.018	50.92
					3.041	58.42
8.	60	10	0	30	3.058	67.69
					3.049	67.1
9.	40	20	10	30	2.931	62.05
					2.908	59.9
10.	50	10	10	30	3.057	66.7
					3.051	66.8

Fig. V-6 Monte-Carlo search for optimal grading of aggregates in composition No. 9

TABLE : V-4

Monte-Carlo search for optimal grading of aggregates with composition no.17 in hot pressing.

Cement content   : 20%
Water/Cement Ratio   : 0.14
Pressing Load   : 10 tonnes
Sample diameter   : 5 cm.
Cured   : 30-35°C in 90% r.h., 24 hrs.
                         : 30-35°C under water, 7 days
Dried   : 110°C, 24 hr.

| No. | grit no.wt% | | | | B.D. [gms/cm^3] | T.S. [Kg/cm^2] |
	10	24	100	- 200		
1.	50	20	0	30	2.957	58.32
					2.939	56.41
2.	60	10	10	20	2.956	64.12
					2.973	65.81
3.	60	20	0	20	2.875	55.12
					2.862	52.45
4.	40	10	20	30	2.848	50.49
					2.828	49.37
5.	50	10	0	40	2.941	52.98
					2.899	52.32
6.	70	0	10	20	2.955	45.73
					2.972	50.49
7.	60	10	0	30	2.941	51.28
					2.923	51.32
8.	40	30	0	30	2.964	56.26
					2.989	59.21
9.	50	20	10	20	2.951	60.86
					2.956	60.08
10.	70	0	0	30	2.872	32.67
					2.893	35.53
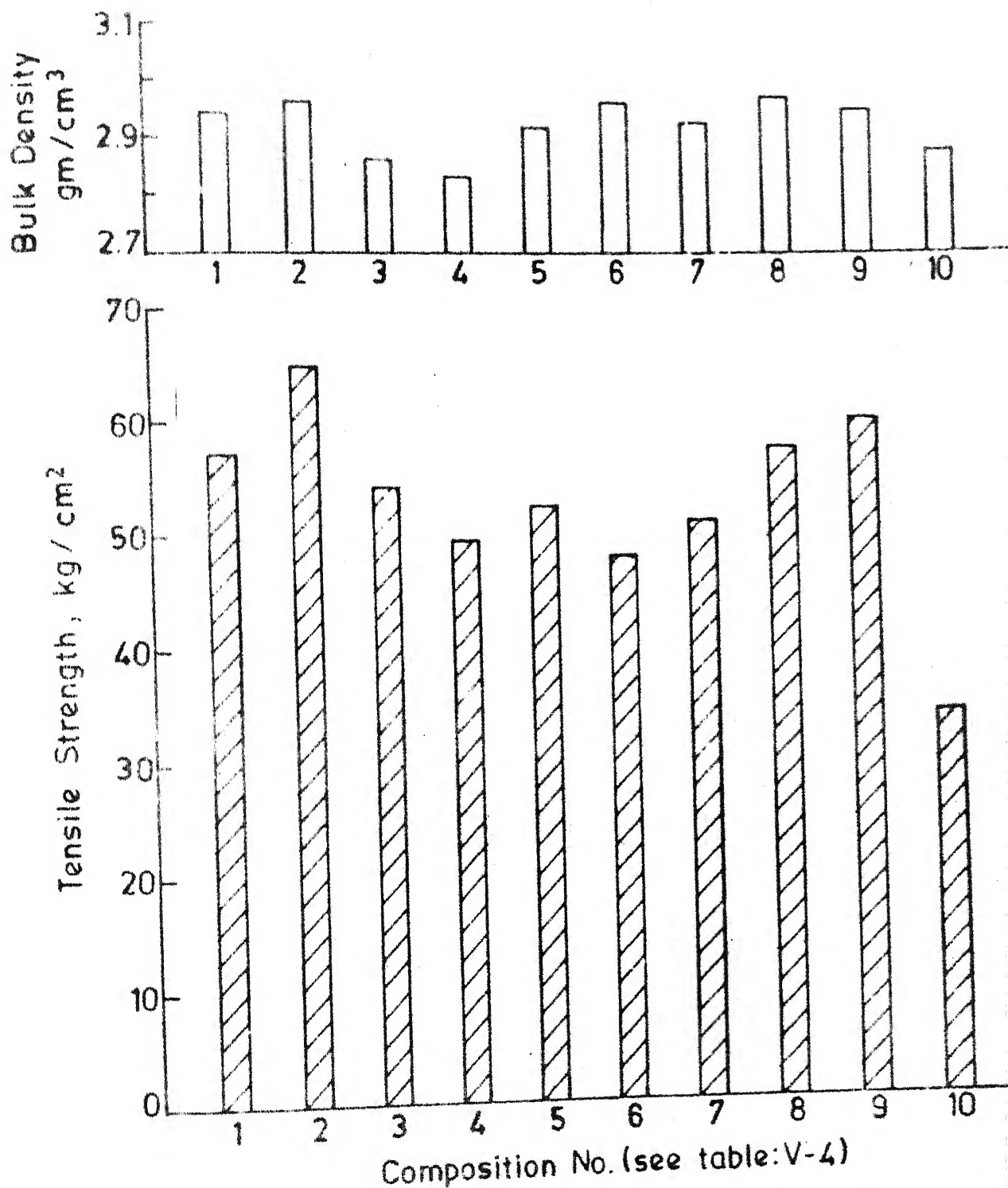
Fig. V-7 Monte Carlo search for optimal grading of aggregates in composition no. 17.

understandable in view of the fact that the original batch-2
composition used in the last series of experiments was in
itself a dense packing mixture and therefore the results are
not unexpected. Any further segnificant improvement would
require higher molding pressure, temperature, and holding
time.

3.   PHOSPHATE BONDED ALUMINA :

In order to compare these results with the
competing chemical bond, namely phosphate bonded alumina
samples were prepared bonded with phosphoric acid (H_3PO_4)
alone and phosporic acid and cromic oxide ($H_3PO_4+CrO_3$). The
compositions were batch-3 pressed under total load of 20
tonnes and batch-4 pressed under total load of 3 tonnes as
shown in table: A-4. The pressed samples were cured at
400,600 and 800°C with 5 hrs. soaking time.

The test results are shown in table: A-4 and
also exhibited in fig: V-8 and fig : V-9. It is seen in
fig : V-8 where data for H_3PO_4 alone is included, that the
strength does not seem to change a great deal between
400 to 800°C. Understandably the strength of samples with
20 tonnes pressing load is more than the strength of samples
with 3 tonnes pressing load, the mean increase being roughly
50% .

It is seen in fig: V-9 that in the case of $H_3PO_4 + CrO_3$ bond at 800^oC the strength of both 20 and 3 tonnes loads are very roughly 50 to 90% higher than the corresponding values for H_3PO_4 only. At 600^oC for 20 tonnes load samples with $H_3PO_4 + CrO_3$ bond in again much superior than the corresponding H_3PO_4 bond. At 400^oC the difference in strength for two bonds are not very large. Overall $H_3PO_4 + CrO_3$ bond is superior to the H_3PO_4 bond and the superiority becomes more marked as the pressing load is increased from 3 tonnes to 20 tonnes and the curing temperature is raised from 400 to 800^oC.

The highest strength obtained is namely about 65 Kg/cm^2 which is still less than the highest strength obtained in hot pressed W.H.A.C. bonded concrete.

CHAPTER -VI

CONCLUSION

On the basis of these experiments and the results obtained thereform , the following conclusions can be drawn:

1. A strong concrete mix based on fused alumina aggregates and high purity white high alumina cement (W.H.A.C.) can be developed with good room temperature as well as, high temperature properties.

2. In cold pressed concrete the effect of molding pressure on tensile strength, decreases as the cement content increases in the range of 5 to 12.5%. Above 12.5% cement content there is virtually no difference going from 10 tonnes to 20 tonnes pressing load.

3. Optimum water/cement ratio for cold pressed concrete is 0.33 at 10 tonnes pressing load.

4. Higher pressing load than 20 tonnes on 5 cm. diameter sample has little effect on bulk density and tensile strength of the cold pressed concret mix.

5. Very strong concrete can be developed by hot pressing at $100^{o}C$ for 10 minutes, with lower water/cement ratio. The best strength value obtained is considerably highe even than the value obtained by bonding the same aggregates with $H_3PO_4 + CrO_3$ and curing at $800^{o}C$ which are the best

process parameters for phosphate bonding.

6. Three promising compositions were :

Composition no. as in T:V-1	Cement Content%	w/c ratio	Pressing Load,tonnes
5	12.5	0.14	20
9	20	0.11	20
17	20	0.14	10

7. Monte-Carlo search for improved gradings of aggregates result in somewhat higher strength for low molding pressure composition [no.17 in T: V-1] and low cement content composition [no.5 in T:V-1]. But for the no. 9 composition having highest strength value, no improvement was obtained.

8. The final compositions are :

Comp. no.as in T:V-1	Grading wt%			
	10 grit	24 grit	100 grit	-200 grit
i) 5	50	10	10	30
ii) 9	40	10	10	40
iii)17	60	10	10	20

i) It can be used where high refractoriness is required, because of its low cement content.

ii) Because of its high strength but high cement content it can be used where abrasion resistance is more important than refractoriness.

iii) It can be used where wear and tear of the die material is to be avoided, because of its low pressing load.

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APPENDIX

TABLE : A-1

Effect of cement content on bulk density and tensile strength of cold pressed concrete.

Composition:

Batch -1	Grit no.		wt%
Fused Alumina	10 10		50
		24	15
		100	15
	- 200		20- W.H.A.C.
W.H.A.C			Rest

Pressing load	:	20 tonnes and 10 tonnes
Water/ cement ratio	:	0.33 for 20 tonnes
	:	0.36 for 10 tonnes
Sample diameter	:	5 cm.
Cured	:	30-35°C in 90% r.h., 24 hr.
	:	30-35°C under water, 7 days
Dried	:	110°C, 24 hr.

TABLE A-3

Effect of molding pressure on bulk density and tensile strength of cold pressed concrete.

Composition:

Batch- 2	Grit no.	wt%
Fushed Alumina	10	50
	24	15
	100	15
	-200	10
W.H.A.C.		10

Sample diameter : 5 cm

Cured : 30-35°C in 90% r.h., 24 hr.

: 30-35°C under water, 7 days

Dried : 110°C , 24 hr.

No.	Pressing load [tonnes]	Water/ Cement Ratio	B.D. [gm/cm^3]	T.S. [Kg/cm^2]
1.	5	0.37	2.933	24.72
			2.912	25.03
2.	7.5	0.37	2.934	25.71
			2.931	27.11
3.	10	0.36	2.945	27.21
			3.014	27.03
4.	15	0.34	3.040	31.28
			3.037	35.02
5.	20	0.33	3.103	35.79
			3.112	39.99
6.	30	0.25	3.126	36.66
			3.126	35.54

TABLE : A -4

Effect of curing temperature and molding pressure on bulk density and tensile strength of phosphote bonded fused alumina bodies.

Composition:

	Grit no.	Batch-3 for 20t load, wt%	Batch-4 for 3 t load, wt%
Fused Alumina	10	50	50
	24	25	10
	100	10	10
	-200	6	20
Bayer's Al_2O_3	72 hrs. wet ground	9	10

Sample diameter : 5 cm.
Pressing Load : 20 tonnes and 3 tonnes
Bond : H_3PO_4 ; 5.2 c.c. for 20 t
         ; 7 c.c. for 3 t.
: $H_3PO_4 + CrO_3$; 7 c.c. for 20 t and 3 t.
Dried : $110^{o}C$, 24 hrs.
Soaking time : 5 hrs.

	H_3PO_4 ; sp. gr: 1.72			$H_3PO_4 + CrO_3$; sp. gr.2.159		
	20 t press.					
Property	$400^{o}C$	$600^{o}C$	$800^{o}C$	$400^{o}C$	$600^{o}C$	$800^{o}C$
B.D.	3.007	3.083	3.053	3.177	3.123	3.088
	3.022	3.062	3.053	3.177	3.134	3.088
T.S.	42.12	41.20	44.00	46.25	57.46	63.86
	43.22	42.34	44.87	42.13	54.38	62.47
	3 t press.					
B.D	2.890	3.002	2.950	2.950	2.920	2.780
	2.870	3.012	2.910	2.930	2.900	2.810
T.S.	36.76	35.79	33.95	37.98	40.28	53.45
	33.76	35.92	33.72	36.89	38.23	54.23

TABLE: A-2

Effect of water/ cement ratio on bulk density and tensile strength of cold pressed concrete.

Composition :

Batch-2	Grit no.	wt%
Fused Alumina	10	50
	24	15
	100	15
	-200	10
W.H.A.C.		10

Pressing load   : 10 tonnes
Sample diameter : 5 cm.
Cured   : 30-35°C in 90% r.h., 24 hr.
                  : 30-35°C under water, 7 days.
Dried   : 110°C, 24 hr.

No.	Water/ Cement ratio	B.D.[gm/cm^3]	T.S. [Kg/cm^2]
1.	0.5	2.780	15.50
		2.741	15.20
2.	0.4	2.820	22.28
		2.882	24.92
3.	0.33	2.945	27.26
		2.901	26.98
4.	0.25	2.942	28.31
		2.962	30.12

Contd ....TABLE: V-1

13.	17.5	0.2	10	2.975	45.68
				2.997	45.16
14.	17.5	0.13	20	2.980	59.20
				3.049	-
15.	12.5	0.13	7.5	2.937	34.72
				2.906	35.20
16.	15	0.14	15	3.031	48.88
				3.013	46.29
17.	20	0.14	10	2.968	61.20
				2.988	60.98
18.	17.5	0.11	25	3.078	59.53
				3.068	59.56
19.	12.5	0.12	20	3.074	48.23
				3.089	46.90

In these series of experiments for each of these compositions, the cement content, w/c ratio and the pressing load was kept constant. Only the grading of the aggregate portions were altered by a Monte-Carlo random search method.

The levels of variation of different grits were as bellow,

Ave. Grit no.	wt%	
10	40,50,60,70	[a]
24	30,20,10, 0	[b]
100	30,20,10, 0	[c]
-200	40,30,20, D	[d]

with the following constraints,

i) $a+b \leq 80$, otherwise the structure would be too coarse and will become not suitable for pressing.

ii) $a+b+d \leq 100$, otherwise the composition become meaningless.

iii) $c = 100 - (a+b+d)$

iv) d includes the cement content which must be deducted when adding the - 200 grit alumina aggregate.

So the procedure followed was as bellow, a and b were picked randomly and checked that their sum

the stable boehmite is formed rather than gibsit .

Next step in this series of experiment was to optimize the grading of the agregates.

B. OPTIMAL GRADING OF AGGREGATES FOR HOT PRESSING:

Three composition from last series of experiments were chosen for optimising the grading of the aggregates for improving the bulk density and tensile strength still further if possible.

The choice of the composition was based on the following conditions,

i) composition number 9 because it gives the highest strength.

ii) composition number 5 which gives the highest strength of all the low cement content compositions.

iii) composition number 17 which gives high strength inspite of low molding pressure.

For convenience the compositions and process conditions are summerized below,

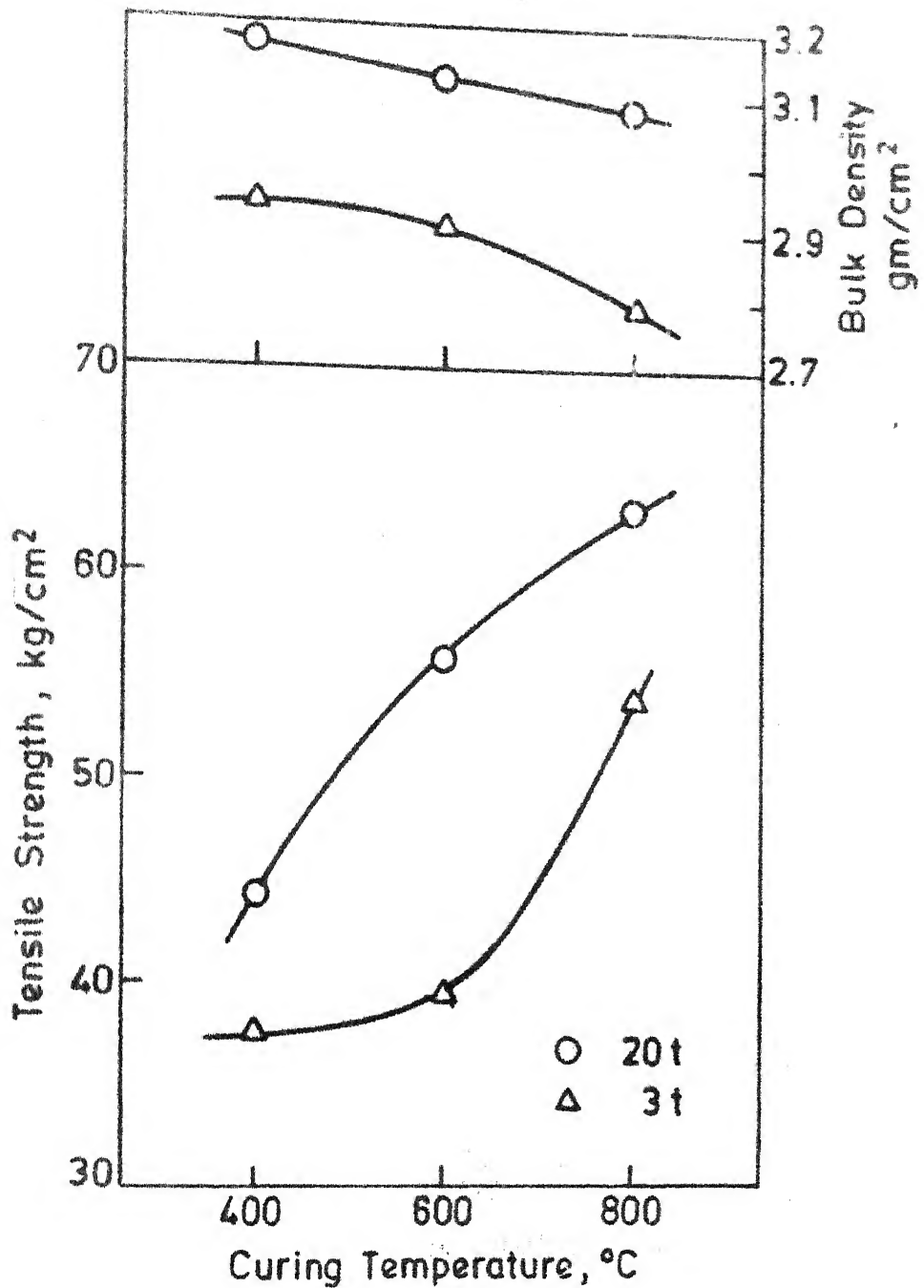| Composition no.	Cement content%	w/c ratio	Pressing load, tonnes
5	12.5	0.14	20
9	20	0.11	20
17	20	0.14	10

Fig. V-9 Effect of curing temperature on bulk density and tensile strength of phosphat bonded fused alumina bonded by $H_3PO_4 + CrO_3$ at two loads.

COMPOSITION: Batch-3 for 20t; Batch-4 for 3t
BOND: $H_3PO_4 + CrO_3$; 7 c.c. for 20t and 3t
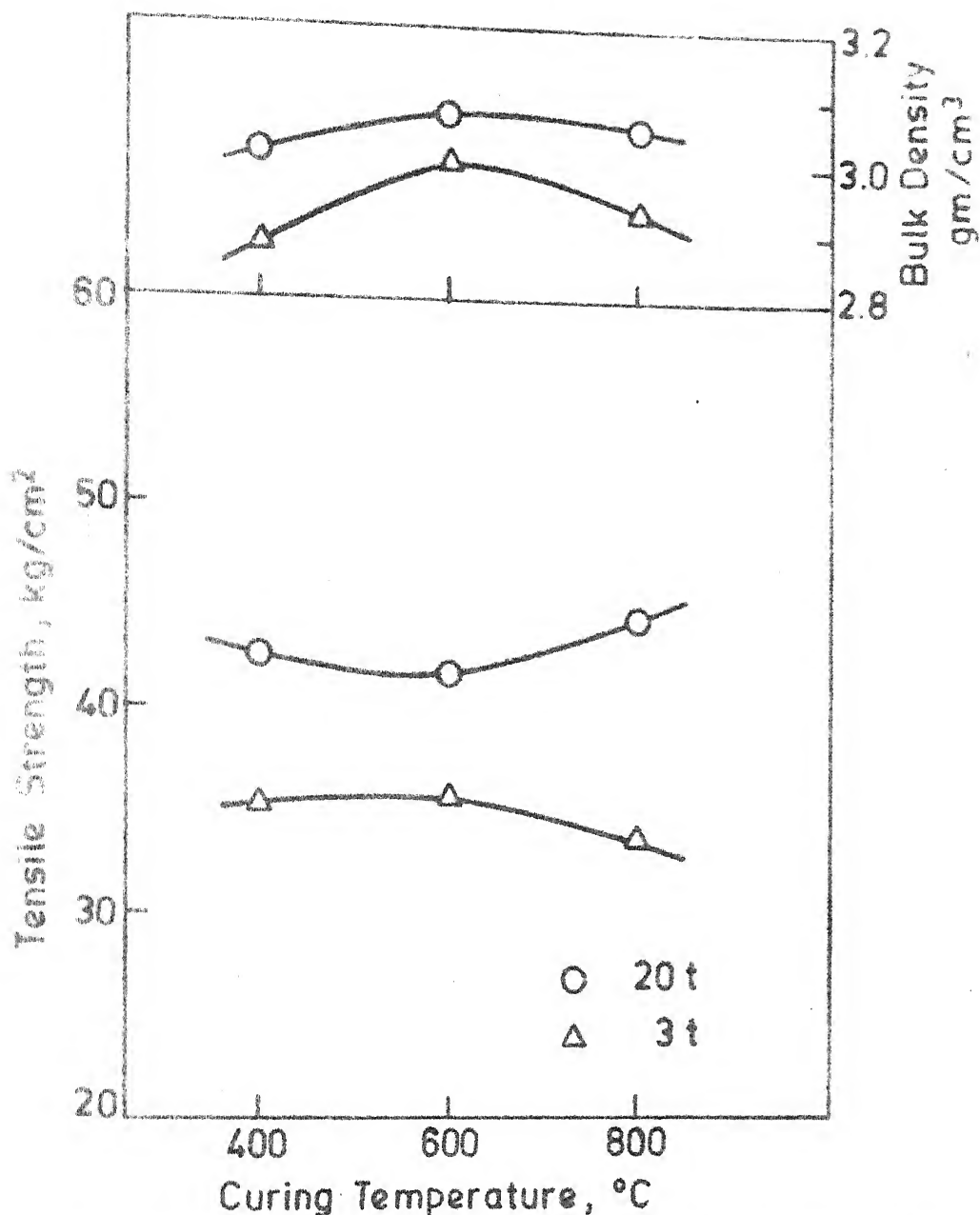SAMPLE DIAMETER: 5 cm
DRIED: 110°C, 24 hr
SOAKING TIME: 5 hr

Fig. V-8 Effect of curing temperature on b.d. and t.s. of phosphate bonded fused alumina bonded by by H_3PO_4 at two pressing loads.

COMPOSITION : Batch-3 for 20t ; Batch-4 for 3t.
BOND : H_3PO_4 ; 5.2 c.c. for 20t ; 7.0 c.c. for 3t
SAMPLE DIAMETER : 5 cm
DRIED : 110°C , 24 hr
SOAKING TIME : 5 hr

No.	20 tonnes B.D., [gm./cm^3]	20 tonnes T.S. [Kg/cm^2]	No.	10 tonnes B.D. [gm/cm^3]	10 tonnes T.S. [Kg/cm^2]	Cement content, %
1.	3.053	29.21	2.	3.062	25.40	5
	3.060	31.49		3.060	29.06	
3.	3.087	31.68	4.	2.860	25.08	7.5
	3.031	31.48		2.980	27.20	
5.	3.103	35.79	6.	2.945	27.21	10.
	3.112	39.99		3.014	27.03	
7.	3.070	44.44	8.	2.965	40.23	12.5
	3.089	39.00		3.099	42.C1	
9.	3.153	42.94	10.	3.029	42.62	15
	3.131	43.01		3.053	43.04	
11.	3.169	45.98	12.	3.042	44.92	20
	3.158	49.19		3.059	48.27	